

(19) Japan Patent Office (JP)

(12) KOKAI TOKKYO KOHO (A)

(11) Unexamined patent publication number Heisei **2-208342**

(43) Publication date: 1990 August 17

(51) Int. Cl. ⁵	ID No.	Office File No.
----------------------------	--------	-----------------

C 08 L 25/00	LED	7445-4J
--------------	-----	---------

71/00 LQK		7921-4J
-----------	--	---------

Examination: Not yet applied for Claims: 1

(Total 7 pages)

(54) Title: Optical disc base

(21) Patent application number: Heisei 1-29175

(22) Application date: 1989 February 8

(72) Inventor: Kenji MANABE

C/o Sumitomo Chemical Industries KK

1-go, 5-ban, Sokai-cho, Niihama-shi, Ehime-ken

(72) Inventor: Masahiro NIWANO

C/o Sumitomo Chemical Industries KK

1-go, 5-ban, Sokai-cho, Niihama-shi, Ehime-ken

(71) Applicant: Sumitomo Chemical Industries KK
33-go, 5-ban, 4-chome, Kitahama, Chuo-ku
Osaka

(74) Agent: Patent attorney Mitsuhiro MOROISHI
and one more person

Text of specifications

1. Title of the invention

Optical disc base

2. Claims

.....

3. Detailed description of the invention

Field of industrial application

The present invention deals with optical disc bases made of resin. In particular, it deals with injection moulded disc bases that have small birefringence and are suitable for magneto-optical recording media.

Existing technology

There have been various suggestions on optical disc bases moulded from resin materials, and many instances of their manufacture. Such bases have different advantages and disadvantages, depending on the material used. US patent 4,373,065 describes an optical recording element made of an optically isotropic resin with practically zero birefringence. This is achieved by mixing two types of completely miscible polymers having just the opposite optical anisotropy, at a ratio that exactly cancels out the optical anisotropy of each polymer.

This US patent also showed that in a system where polyphenylene ether and polystyrene are used as the polymers with the opposite optical anisotropy, if the composition of the mixture where the anisotropies are exactly cancelled out is used to make a film, the film does not develop birefringence even when drawn and oriented by applying stress. Tokkaisho (unexamined Japanese patent publication number Showa) 62-240901 claims that optical elements with very small birefringence can be produced even by injection moulding from a material with a polymer component consisting mainly of an aromatic vinyl monomer and a polyphenylene ether component, by suitably selecting the molecular weight and composition of the two components.

Problems to be solved by the new invention

Optical disc bases are required to have very low optical anisotropy, which manifests as birefringence, while at the same time having a large Carrier/Noise ratio (C/N ratio) at the time of recording and playback.

The above-referred US patent 4,373,065, however, does not claim that if a polymer having this composition were made into optical disc bases by injection moulding, such optical materials would have very low birefringence. In fact, the

birefringence is not low for all of such a polymer composition. Tokkaisho 62-240901 does not explicitly demonstrate that injection moulded optical disc bases made from the specified polymer composition would make the birefringence of the disc base low in all the three dimensions.

Attempts are currently being made to make plastic disc bases for erasable and rewritable magneto-optical discs.

For reading the information recorded on such magneto-optical systems, a polarised laser beam is focussed on the recording medium with the help of a lens and the slight rotation of the polarised light in the reflected laser beam because of the Kerr effect is detected. Therefore, we have to use an optical disc base that does not easily add birefringence to the incident light that comes in a diagonal direction.

Against the above background, the present invention aims at providing optical disc bases with advantageous properties even when injection moulded. These include low birefringence for perpendicularly incident light and diagonally incident light, large C/N ratio, high heat resistance, good balance of mechanical strengths, and superior dimensional stability.

The solution

In short, the present invention deals with an optical disc base prepared by injection moulding of a resin composition comprising 50-70 parts by weight of a polymer component consisting mainly of an aromatic vinyl monomer and 50-30 parts by weight of a polyphenylene ether component, that satisfies the conditions listed below:

$$|N_x - N_y| \leq 5 \times 10^{-5}$$

$$|N_x - N_z| \leq 5 \times 10^{-4}$$

$$|N_y - N_z| \leq 5 \times 10^{-4}$$

Here, N_x is the average refractive index along the diameter direction of the disc base, N_z is the average refractive index along a direction perpendicular to the plane of the disc base, and N_y is the average refractive index along a direction perpendicular to the directions of both N_x and N_z . ###

When N_x , N_y and N_z have the meanings given above, $|N_x - N_y|$ is the birefringence for the light incident perpendicularly to the plane of the disc base, $|N_x - N_z|$ and $|N_y - N_z|$ are respectively indices that represent the birefringence and correlation for the light incident diagonal to the plane of the optical disc, in the optical disc bases injection moulded from the resin composition.

In the new invention, $|N_x - N_y|$ must be 2×10^{-5} or less, preferably less than 1×10^{-5} .

If $|N_x - N_y|$ exceeds 2×10^{-5} , the retardation when the laser beam enters perpendicular to the disc base would be more than 24 nm for discs of thickness 1.2 mm, which is not desirable.

$|N_x - N_z|$ and $|N_y - N_z|$ should both be not more than 2×10^{-4} . If $|N_x - N_z|$ or $|N_y - N_z|$ exceeds 2×10^{-4} , the average retardation of the laser beam that enters from a direction inclined 30° from the perpendicular to the plane of the disc exceeds 20 nm, while C/N ratio is lowered at the same time, which should be avoided.

In the new invention, "a resin composition comprising a polymer component, having an aromatic vinyl monomer as its main constituent, and a polyphenylene ether component" means a mixture of these two types of polymers, a block copolymer or graft copolymer consisting of these two polymer components, or mixtures of such copolymers.

The polymer component having an aromatic vinyl monomer as its main constituent can be a homopolymer of an aromatic vinyl monomer or a copolymer having at least 50 wt.% of the aromatic vinyl monomer units. Examples of aromatic vinyl monomers are styrene, α -methyl styrene, m-methyl styrene, p-methyl styrene, o-chlorostyrene, m-chlorostyrene, p-chlorostyrene, m-bromostyrene and p-bromostyrene. Among these, styrene is particularly suitable.

Examples of monomers that can be copolymerised with the above-listed type of aromatic vinyl monomers are unsaturated nitriles like acrylonitrile and methacrylonitrile; alkyl esters of methacrylic acid like methyl methacrylate, n-propyl methacrylate, isopropyl methacrylate, n-butyl methacrylate, and cyclohexyl methacrylate; alkyl acrylates like methyl acrylate, ethyl acrylate, propyl acrylate and butyl acrylate. Methacrylic acid, acrylic acid, maleic anhydride, anhydrous citraconic acid, N-methyl maleimide and N-phenylmaleimide are additional examples.

These monomers can be used for copolymerisation either singly or in combinations. The combinations and their proportions in the copolymer may be adjusted in such a way that the transparency of the final resin composition comprising a copolymer, having an aromatic vinyl monomer as its main constituent, and a polyphenylene ether component, is not adversely affected.

The suitable amount of the aromatic vinyl monomer in the monomer mixture is 50 wt.% or more. Less than 50 wt.% of this component is not suitable because then the resin prepared will have too high a hygroscopicity.

The polymer component having the aromatic vinyl monomer units as its main constituent should preferably have a melt flow rate (MFR) of 0.5-200, more preferably 2-100, at 230°C under 3.8 kg load.

An MFR of more than 200 is not desirable because this would adversely affect the mechanical strength of the material. Less than 0.5 is also not suitable because this makes it difficult to lower the birefringence.

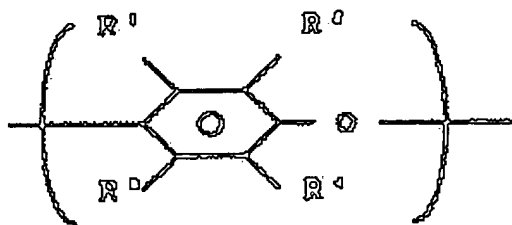
The polymer component having the aromatic vinyl monomer units as its main constituent can be prepared by bulk polymerisation, suspension polymerisation, emulsion polymerisation or solution polymerisation, using a radical initiator. However, bulk or suspension polymerisation is preferable, considering the productivity and the need to prevent intermixing of impurities.

Examples of radical initiators that can be used are peroxides like lauroyl peroxide, benzoyl peroxide, di-*tert*-butyl peroxide, dicumyl peroxide, etc and azo compounds like 2,2'-azobis isobutyronitrile and 1,1'-azobis(1-cyclohexane carbonitrile).

In addition to this, for regulating the molecular weight, a chain transfer agent like *tert*-butyl, n-butyl, n-octyl, n-dodecyl and tertiary dodecyl mercaptans may be added if needed.

The polymerisation may be normally carried out in the temperature range 50-150°C.

The polyphenylene ether component in this invention is a polymer with repeating units that can be represented by the general formula given below.



(Here, R^1 to R^4 are hydrogens, halogens or hydrocarbon groups)

Such polyphenylene ethers are polymers prepared by oxidative coupling of phenol monomers. They can be easily prepared by a well-known method where a copper or a manganese catalyst is used (like the methods described in Japanese patents 36-18692 and 47-36518).

Practical examples of such polyphenylene ethers are poly(2,6-dimethyl-1,4-phenylene)ether, poly(2-methyl-6-ethyl-1,4-phenylene)ether, poly(2-methyl-6-propyl-1,4-phenylene)ether, poly(2,6-dipropyl-1,4-phenylene)ether, poly(2-methyl-6-bromo-1,4-phenylene)ether, etc. Among these, poly(2,6-dimethyl-1,4-phenylene)ether is particularly suitable.

Polyphenylene ethers commonly used as engineering plastics can also be used, but the ones with smaller molecular weight are more suitable.

The suitable range of mean molecular weight of the polyphenylene ether is 0.1-1.0, preferably 0.3-0.5, expressed in terms of the limiting viscosity (in chloroform, measured at 25°C). If this viscosity is less than 0.3, the mechanical strength of the optical disc bases prepared from the resin would be comparatively small. The limiting viscosity should preferably not exceed 0.5 to keep the birefringence for light entering the disc perpendicularly, and particularly for light entering diagonally, small.

Melt-mixing or solution mixing can be used for preparing the resin composition in this new invention by mixing the polymer having an aromatic vinyl monomer as the main constituent and the polyphenylene ether component.

Melt-mixing may be done under a high shearing force in a mixing device such as an extruder, Banbury mixer, kneader- blender, heated rollers, etc at or above the glass transition point of the polyphenylene ether.

The mixing must be done to the extent that both the polymers are dispersed to units of about $1\ \mu$ or less. Preferably, the mixing may be continued up to a state of molecular level mixing. Whether molecular level mixing has been achieved or not can be easily found out by checking whether the glass transition point of the mixture has become one single value.

To achieve thorough and satisfactory mixing, a higher mixing temperature, a longer mixing time, or a higher shearing force, etc may be employed.

In melt-mixing, a small amount of organic solvent may be added as a plasticiser to make the mixing easier by lowering the glass transition point of the polymers. The organic solvents listed for use in solution mixing are applicable here also. After the mixing, this organic solvent may be evaporated off.

In solution mixing, the polymers are dissolved in an organic solvent to prepare a solution of at least 1 wt.%. Then, after obtaining a uniform mixture through stirring and blending, the organic solvent may be evaporated off. Alternatively, a poor solvent

of the two polymer components may be added to the uniform mixture prepared, to allow the precipitation of the polymer mixture.

Examples of suitable organic solvents are chloroform, methylene chloride, ethylene chloride, toluene, benzene and chlorobenzene. Examples of poor solvents are methanol, ethanol, propyl alcohol, n-hexane and n-pentane.

Block copolymers or graft copolymers consisting of the polymer component-the main constituent of which is the aromatic vinyl monomer, and the polyphenylene ether component can be obtained by polymerising one of the polymer components in the presence of the other polymer component. To be more specific, one of the methods suggested in Japanese patents 42-22069, 47-1210, 47-47862 and 52-38596 may be employed. The graft copolymer or block copolymer is prepared by polymerising the monomers, consisting mainly of the aromatic vinyl monomer units, in the presence of the polyphenylene ether. Alternatively, the phenol monomer is polymerised by oxidative coupling in the presence of the polymer component-the main constituent of which is the aromatic vinyl monomer units, to prepare the graft polymer or graft copolymer.

Here, 50-70 wt.% of the polymer component having the aromatic vinyl monomer unit as its main constituent, and 50-30 wt.% of the polyphenylene ether component are used in the resin composition.

If the polyphenylene ether component is less than 30 wt.% or more than 50 wt.%, the birefringence of the optical disc bases prepared from such resin would not be

sufficiently low for diagonally incident light. Moreover, with less than 30 wt.% of this component, the heat resistance also would not be satisfactory.

Injection moulding is the most suitable method of moulding the newly invented optical disc bases because it is the most commonly used method of producing mouldings from resin compositions and the method has high productivity.

Here, injection moulding means introducing, under pressure, the molten resin into a closed metal cavity and cooling to solidify it.

Vacuum suction into the mould or the injection-compression method, where the volume of the metal cavity is shrunk during the moulding, may also be used in combination.

While making the newly invented optical disc bases by injection moulding, it is preferable to have the molten and plasticised resin at 270-350°C. A temperature higher than 300°C is preferable for meeting the requirements of the new invention. Here, the temperature of the resin means the temperature of the resin inside the injection cylinder where the resin has become molten and plasticised because of external heating by heaters, etc installed in the injection moulding machine, and also by the heat generated by the shearing caused by rotation of the screw.

If the resin temperature is less 270°C, the optical disc base prepared becomes unsuitable because $|N_x - N_y|$ becomes more than 2×10^{-5} . On the other hand, a resin temperature higher than 350°C causes decomposition of the resin, which leads to defects like burn marks, silvering, etc, considerably increasing the bit errors in the optical disc.

The suitable mould temperature used in injection moulding is the glass transition point minus 40°C ($T_g-40^{\circ}\text{C}$) or higher but not higher than $T_g-10^{\circ}\text{C}$. The mould temperature here means the surface temperature of the mould cavity just before injection of the resin. If the mould temperature, is below $T_g-40^{\circ}\text{C}$, the transferability of the fine grooves on the stamper of the mould surface becomes poor. On the other hand, if the mould temperature exceeds $T_g-10^{\circ}\text{C}$, the mould release property gets adversely affected.

In the new invention, the suitable rate of injection in the injection moulding is 5 g/sec to 100 g/sec, for making a 1.2 mm thick disc base. A more preferable rate is 10-60 g/sec. With less than 5 g/sec, the temperature distribution in the resin inside the mould becomes too large, which in turn increases the optical anisotropy of the disc base prepared. More than 100 g/sec is also not suitable because then the shearing heat raises the temperature of the resin too much which causes resin decomposition, which sharply increases the bit error when it is made into an optical disc.

The optimum extrusion rate can be determined after finalising the resin composition, by making several trial runs.

Examples

The new invention will now be explained in greater detail with the help of some examples. But what is described here does not in any way limit the scope of the invention.

“Parts” and “%” mentioned in these examples are all on weight basis. In these examples, the physical properties were determined by the methods specified below.

. $|N_x-N_y|$, $|N_z-N_y|$ and $|N_z-N_x|$: The retardation was measured with an ADR-300B (Oak Seisakusho) using an 830 nm laser beam shone on the base from a direction perpendicular to it and at 15 and 30° inclinations from the perpendicular, at

positions 30, 40 and 50 mm from the centre of the disc, and the values were calculated.

. Water absorption : Equilibrium water absorption was measured in distilled water of 23°C, as per ASTM D-570.

.Light transmittance : Light transmittance was measured at 800 nm, with a 1.2 mm thick sample using an Autorecording Spectrophotometer 330 (Hitachi Ltd.).

.Bending property: Measured as per ASTM D-790.

.Heat resistance: Was expressed in terms of glass transition point, measured by the linear expansion coefficient method.

.Limiting viscosity of the polymer: Measured in chloroform solvent at 25°C using a Ubbelohde viscometer and calculated.

.Blending and pelletisation were done in a biaxial extruder (Tex 30-30AW-2V, Nihon Seiko Co. Ltd.)

.The extrusion moulding machine used was Neomat 150/75 (75 tonne, Sumitomo Heavy Industries Ltd) and a disc mould for 130 mm diameter and 1.2 mm thick mouldings was used.

.Conditions of recording on the magneto-optical discs

rpm:	1,800
Recording frequency:	1 MHz
Duty:	50%
Recording power:	6 mW
Recording magnetic field:	350 Oe
Recording time:	10 sec (for about 300 tracks)

C/N: Measured at the position of disc radius 30 mm using a magneto-optical disc evaluation device OMS-1000 (Nakamichi Co.).

Example 1

Following the method described in Example 2, No. 9 of Japanese Patent 47-86518, 2,6-xylenol was polymerised using manganese chloride and ethanolamine as catalysts to obtain poly(2,6-dimethyl-1,4-phenylene)ether with limiting viscosity 0.46 (in chloroform, 25°C).

The polyphenylene ether resin prepared above and a polystyrene (Espride® 4-62A, Nihon Polystyrene) were mixed at the ratio shown in Table 1, kneaded and pelletised in an extruder.

These pellets were extrusion moulded at cylinder temperature 320°C and mould temperature 110°C to prepare grooved optical disc bases of diameter 130 mm and thickness 1.2 mm, with grooves of pitch 1.6 μ .

The physical properties of the discs are shown in Table 1.

Reactive sputtering was applied to the disc bases prepared above under the reduced pressure of 5×10^{-3} Torr in an argon-nitrogen gas mixture to form an 850 Angstrom thick silicon nitride film with refractive index 2.0.

Then, a 900 Angstrom thick Tb Fe Co magnetic layer was deposited by the sputtering following the standard method. An 850 Angstrom thick silicon nitride layer was then made over the magnetic layer by the same method as mentioned earlier to prepare magneto-optical discs having a base/silicon nitride/Tb Fe Co/silicon nitride layer structure. The performance (C/N ratio) of these magento-optical discs was evaluated. The results are given in Table 1.

Examples 2-4

Except for using the different mixing ratios of poly(2,6-dimethyl-1,4-phenylene)ether and polystyrene as shown in Table 1, all other steps were done exactly as in Example 1. The results are given in Table 1.

Comparative Example 1

Disc bases were moulded exactly as in Example 1 but using optical disc grade polycarbonate (AD5503, Teijin Chemicals) and evaluated as in Example 1. The results are given in Table 1.

Table 1

		Example 1	Example 2	Example 3	Example 4	Comparative Example 1
Compo- sition	Polyphenylene ether wt. %	40	42	35	47	
	Polystyrene wt/ %	60	58	65	53	
30*	$ N_x-N_y \times 10^5$	0.2	0.3	0.2	0.3	0.5
	$ N_y-N_z \times 10^4$	1.0	1.2	1.0	1.3	2.5
	$ N_x-N_z \times 10^4$	1.3	1.5	1.2	1.6	6.1
40*	$ N_x-N_y \times 10^5$	0.1	0.2	0.1	0.2	0.4
	$ N_y-N_z \times 10^4$	1.0	1.2	0.9	1.2	2.8

	$ N_x - N_z \times 10^4$	1.2	1.4	1.1	1.5	5.9
50*	$ N_x - N_y \times 10^5$	0.1	0.1	0.1	0.1	0.4
	$ N_y - N_z \times 10^4$	1.0	1.0	1.0	1.1	2.6
	$ N_x - N_z \times 10^4$	1.2	1.1	1.2	1.2	5.7
Light transmittance %		88	87	89	87	90
Water absorption %		0.1	0.1	0.1	0.1	0.35
Tg °C		141	143	135	152	146
Bending strength kg/cm ²		1210	1320	1280	1350	1060
C/N ratio dB		54	55	55	55	48

* : Distance (mm) from the disc centre

Effectiveness of the invention

The newly invented optical disc base has low hygroscopicity, good dimensional stability, and good balance of heat resistance and mechanical strength, besides having small birefringence in three dimensions. Therefore, it is not only suitable as the base for ordinary optical discs but also for magneto-optical discs, where a high C/N can be achieved.

An additional advantage of the new invention is that it provides a method of producing mouldings of low birefringence even by the injection moulding method, which is generally considered to increase birefringence.

Transinfo notes:

2. Kindly check parts marked in orange. Their translation is uncertain or the original

is not legible

1. Parts where the source text appears to have errors are marked in pink.